

## Hydroxyl contents of accessory minerals in mantle eclogites and related rocks

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### ABSTRACT

Infrared spectra in the region 3000 to 3800  $\text{cm}^{-1}$  have been measured on coesite, kyanite, sanidine, and rutile occurring in inclusions of eclogite, and on garnet and spinel in an inclusion of alkremite from South African kimberlites. Coesite and sanidine contain virtually no  $\text{H}_2\text{O}$  or OH, although one sanidine crystal was observed to contain abundant  $\text{H}_2\text{O}$ , apparently as fluid inclusions of molecular  $\text{H}_2\text{O}$ . Kyanite contains significant OH, consistent with previous results for similar samples. Corundum gives no indication of the presence of  $\text{H}_2\text{O}$  or OH. Rutile containing significant amounts of Fe, Cr, Al, and Nb also contains significant amounts of structural OH. Garnet in alkremite contains small amounts of molecular  $\text{H}_2\text{O}$ , whereas the spinel coexisting with this garnet gives no indication of  $\text{H}_2\text{O}$  or OH, indicating that the spinel structure is not able to accommodate significant amounts of OH. Ammonium contents of all samples were less than the detection limit (about 10 ppm).

### INTRODUCTION

$\text{H}_2\text{O}$  and OH in mantle minerals are of importance because their presence can have a major effect on melting relations and other phase transitions in mantle rocks. Further, understanding the mechanisms and limits of OH substitution in nominally anhydrous mantle phases may lead to a predictive capability for  $\text{H}_2\text{O}$  contents of a host of high-pressure phases.

Hydroxyl substitution in nominally anhydrous phases may occur by several mechanisms. However, because there are almost no minerals in which OH (or F) bond directly to Si (a notable exception is thaumasite,  $\text{Ca}_3\text{Si}(\text{OH})_6\text{CO}_3\text{SO}_4 \cdot 12\text{H}_2\text{O}$ ) or to cations of similar or higher charge, it is likely that Si avoidance will be maintained to even trace levels of substitution. Therefore, in orthosilicates OH will most likely substitute either at O sites not bonded to Si, or as an  $(\text{OH})_4$  tetrahedron substituting for the  $\text{SiO}_4$  tetrahedron as in hydrogarnet. Recognition of the former mechanism led to the prediction that wadsleyite ( $\beta\text{-Mg}_2\text{SiO}_4$ ) might be a significant host for OH in the transition zone of the mantle (Smyth, 1987). Spectroscopic measurements have since confirmed that OH or  $\text{H}_2\text{O}$  occurs in this phase (McMillan et al., 1987). The hydrogarnet substitution is common in garnets, and Si-free hydrogrossular has been synthesized and studied (Lager et al., 1987). Aines and Rossman (1984) demonstrated that minor OH contents are commonly found in mantle-derived garnets. This mechanism may also occur in other orthosilicates in which all O is bonded to Si but is not likely to occur in silicates with higher polymerization of the silicate groups, because these would require Si-OH bonds.

The upper mantle, as represented by inclusions in kimberlites and alkali basalts, is composed of two major, distinct, and separate suites of rocks: lherzolites and eclogites, plus minor amounts of exotic types such as garnetites, clinopyroxenites, alkremites (garnet-spinel rocks), and MARIDs (mica-apatite-rutile-ilmenite-diopside). The suites of inclusions in both basalts and kimberlites are dominated by olivine-rich rocks of the lherzolite suite, which are typically highly depleted in basaltic component. These are usually 60–90% olivine, 10–30% orthopyroxene, 5–20% low-Na clinopyroxene and <5% spinel or garnet. This suite is usually free of nominally hydrous phases.

By contrast, the eclogite suite is virtually olivine-free and exhibits a much greater range of mineral compositions and primary accessory phases. The most common eclogites contain just two primary phases, garnet and clinopyroxene, but the garnets are more calcic, and the pyroxenes much more sodic, than garnets and pyroxenes, respectively, of the lherzolite suite. Kyanite may also occur in eclogites with bulk compositions relatively rich in Ca and Na. Primary accessory phases in mantle eclogites include coesite, corundum, sanidine, rutile, phlogopite, amphibole, graphite, and diamond. In addition to the hydrous phases, some of the anhydrous accessory phases have already been shown to contain small amounts of hydrous components either as structurally bound OH groups (kyanite, Beran and Goetzinger, 1987) or as microscopic inclusions of hydrous phases (diamond, Navon et al., 1988).

In order to constrain the OH contents of the eclogite suite of rocks, especially the more extreme, accessory-bearing compositions, we have measured infrared (IR)

TABLE 1. Electron microprobe analyses in weight percent

Mineral sample	Coesite SRV-1	Kyanite SRV-1	Sanidine SRV-1	Corundum JJG-2108	Rutile EJB-86	Phlogopite SRV-16	Garnet SFS-3	Spinel SFS-3
SiO <sub>2</sub>	99.56	36.82	66.63	0.00	0.00	43.40	41.84	0.00
TiO <sub>2</sub>	0.00	0.03	0.00	0.03	95.24	0.57	0.18	0.18
Al <sub>2</sub> O <sub>3</sub>	0.05	62.63	16.77	99.78	0.68	12.07	23.67	64.26
Cr <sub>2</sub> O <sub>3</sub>	0.00	0.06	0.02	0.07	1.16	0.26	0.11	0.87
Nb <sub>2</sub> O <sub>5</sub>	n.d.	n.d.	n.d.	n.d.	1.86	n.d.	n.d.	n.d.
FeO	0.02	0.15	0.05	0.08	0.67	2.66	9.68	13.20
MgO	0.00	0.05	0.00	0.04	0.13	25.82	16.19	20.05
MnO	0.00	0.00	0.00	0.00	0.04	0.01	0.31	0.05
CaO	0.00	0.00	0.00	0.00	0.00	0.00	8.72	0.04
BaO	n.d.	n.d.	0.12	n.d.	n.d.	n.d.	n.d.	n.d.
Na <sub>2</sub> O	0.03	0.00	0.26	0.00	0.00	0.15	0.08	0.02
K <sub>2</sub> O	0.00	0.00	16.79	0.00	0.00	8.90	0.00	0.00
F	n.d.	n.d.	n.d.	n.d.	n.d.	0.24	n.d.	n.d.
Cl	n.d.	n.d.	n.d.	n.d.	n.d.	0.34	n.d.	n.d.
Total	99.66	99.74	100.52	100.01	99.78	94.42	100.78	98.73

Note: n.d. = not determined.

absorption spectra over the region 3000 to 3800 cm<sup>-1</sup> of some of the more abundant primary accessory phases in the eclogite suite. Also, ammonia is a possible volatile component that may be accommodated in the crystal structures of K-bearing phases, phlogopite and sanidine. The ammonia contents of these phases may also be constrained from these IR data. Further, the crystal structure of coesite from one of the samples has been refined by neutron diffraction methods as a reference structure (Smyth et al., 1987) and used to test models of the SiO bond in minerals, and a knowledge of its OH content may be relevant to these studies.

### EXPERIMENTAL METHODS

Room-temperature infrared spectra were obtained with a Nicolet 60SX FTIR at 2-cm<sup>-1</sup> resolution. Polished, self-supporting sample plates were mounted over pinhole apertures to define the viewing area through the sample. Typical viewing areas were 100 to 600 μm in diameter.

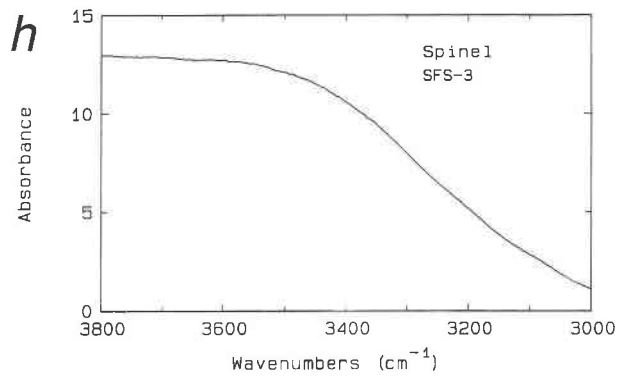
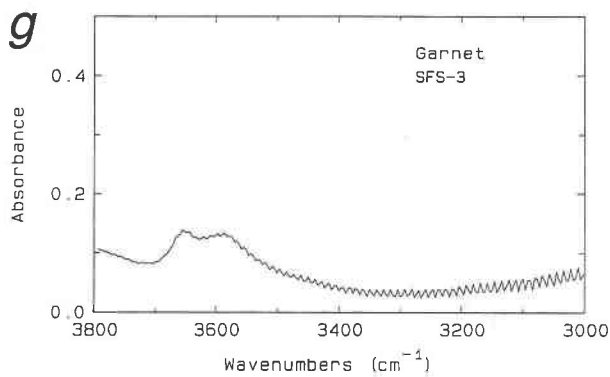
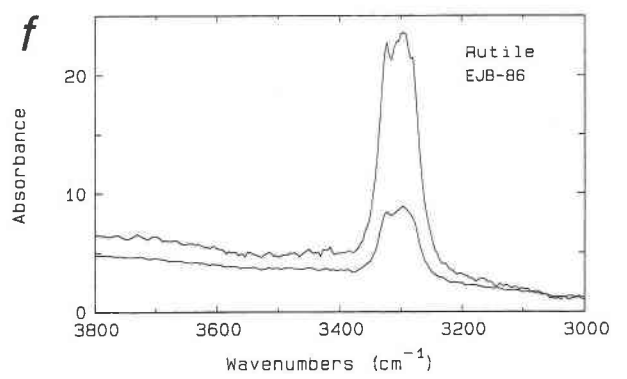
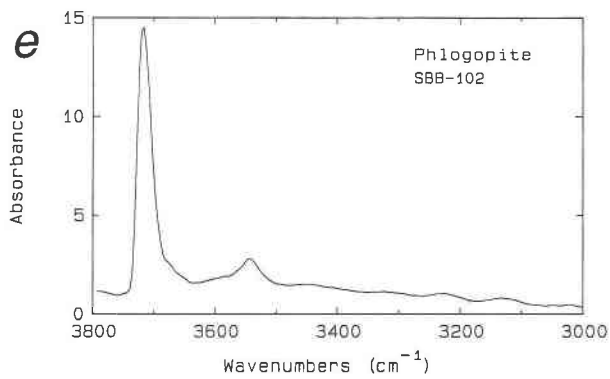
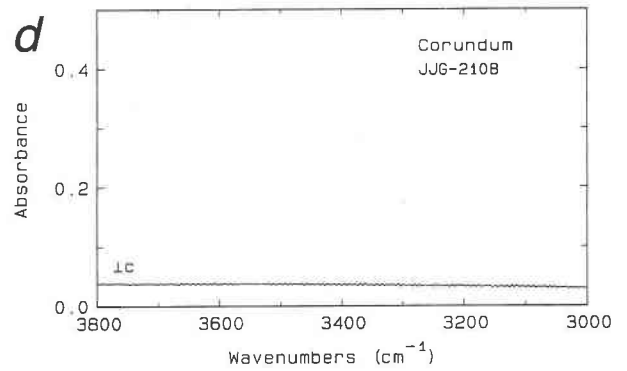
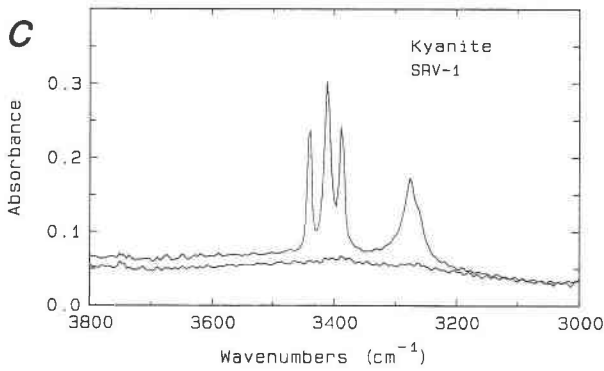
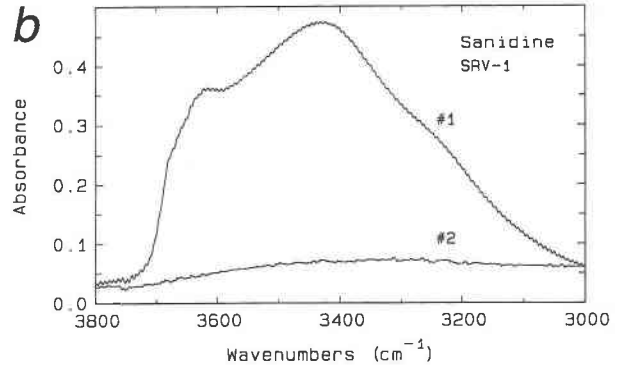
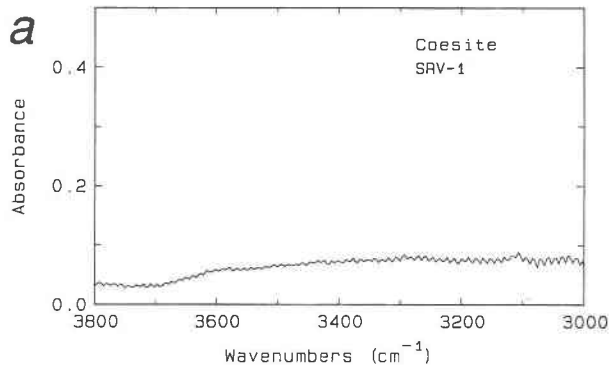
### RESULTS AND DISCUSSION

**Coesite.** Coesite has been reported to occur as an equilibrium phase in kyanite eclogites and grosspydites from South Africa (Smyth and Hatton, 1977; Schulze and Helmstaedt, 1988) as well as from other localities. Three single crystals of coesite from sample SRV-1, a coesite grosspydite from the Roberts Victor Mine, were selected for IR spectroscopic examination and prepared as double-polished grains with thicknesses of 46, 131, and 260 μm. The crystal structure of this natural coesite has been determined by X-ray and neutron single-crystal diffraction methods (Smyth et al., 1987). The composition determined by electron microprobe analysis is given in Table 1.

The IR spectrum of the crystal of coesite 46 μm thick gave no indication whatever of OH or H<sub>2</sub>O. However, the thickest crystal (Fig. 1a) did give some indication of broad band molecular H<sub>2</sub>O. Because open fractures were evident in the thickest crystal, we believe that the molec-

ular H<sub>2</sub>O spectrum represents hydrous material residing in the fractures. For the dry crystal, we estimate the detection limit at 0.003 absorbance units, which would correspond to 0.004 wt% OH (40 ppm) if the H were present in sharp band OH such as in the spectrum of kyanite (see below). Although the corresponding detection limit for broad band H<sub>2</sub>O such as occurs in synthetic quartz (Aines et al., 1984) is more difficult to estimate, we place it at about 0.01 wt% H<sub>2</sub>O. Our sensitivity toward H is not as high as it is in the case of natural and synthetic quartz, where we can use samples several millimeters to centimeters thick. Our sensitivity is necessarily directly pro-

Fig. 1. Infrared absorption spectra in the region 3000 to 3800 cm<sup>-1</sup> for accessory phases in mantle eclogites and for garnet and spinel from a mantle-derived alkremite. All spectra normalized to a thickness of 1000 μm. (a) Coesite from grosspydite SRV-1, actual sample thickness 261 μm: no evidence of molecular H<sub>2</sub>O or OH. (b) Sanidine from grosspydite SRV-1: spectrum no. 1 is from a slightly turbid sample (thickness 442 μm) showing a broad absorbance pattern consistent with molecular H<sub>2</sub>O in fluid inclusions; spectrum no. 2 is from another crystal (thickness 271 μm) showing absence of broad absorbance feature and no evidence of ammonia. (c) Kyanite from sample SRV-1: two spectra from the same crystal, 217-μm thick, polarized at 0° and 90°. Polarized IR absorbance is due to structural OH. (d) Corundum from eclogite JJG-2108 from Bellsbank, thickness 315 μm: no evidence of H<sub>2</sub>O or OH. (e) Phlogopite SBB-102 from Bobbejaan (Bellsbank), thickness 33 μm: spectrum corrected for glass and epoxy; no evidence of ammonia. (f) Rutile from Roberts Victor eclogite EJB-86, sample thickness 46.4 μm: spectra corrected for glass and epoxy, two polarized spectra at 0° and 90° relative show evidence of structural OH. (g) Garnet from alkremite SFS-3, thickness 250 μm: shows a small feature due to molecular H<sub>2</sub>O, probably in fractures or inclusions, but no evidence of OH. (h) Spinel from alkremite SFS-3, thickness 31 μm: shows broad absorbance feature due to Fe<sup>2+</sup>, but no evidence of OH or molecular H<sub>2</sub>O.



portional to the path length through the sample, which, in the case of coesite, is quite limited. The observed absence of OH in this sample means that the conclusions of Smyth et al. (1987) concerning the bond length vs. angle relations at 10 K are not complicated by structural effects of OH, as these authors presumed.

**Sanidine.** Sample SRV-1 (coesite grosspydite) also contains primary sanidine (Smyth and Hatton, 1977), which has the highest degree of Al-Si disorder of any natural or synthetic feldspar yet measured (Scambos et al., 1987), consistent with its high temperature of primary phase equilibration (Wohletz and Smyth, 1984). The composition is nearly pure  $KAlSi_3O_8$  (Table 1). The crystals typically appear somewhat turbid in thin section.

A single crystal approximately  $850 \times 750 \mu\text{m}$  and  $442 \mu\text{m}$  thick was prepared for IR examination. An unpolarized spectrum in the region  $3000$  to  $3800 \text{ cm}^{-1}$  is in Figure 1b as spectrum 1. The broad absorption band and the turbidity are both most likely due to molecular  $\text{H}_2\text{O}$  in fluid inclusions in the sample.

To test whether the broad absorption band in the first crystal was the result of structurally incorporated  $\text{H}_2\text{O}$  or fluid inclusions, a second crystal, approximately  $450 \times 600 \mu\text{m}$ , was oriented crystallographically and polished on two parallel faces normal to  $c$  to a thickness of  $271 \mu\text{m}$  for IR examination. Two polarized spectra were obtained using a  $\text{LiIO}_3$  crystal polarizer, one of which is shown in Figure 1b as spectrum 2. The spectra showed very little of the broad absorption band observed in the first spectrum. Furthermore, there were no polarization effects, which would be expected for structural  $\text{H}_2\text{O}$ . We conclude that the broad band was due to water in microscopic fluid inclusions. Using a spectrum from liquid  $\text{H}_2\text{O}$  between glass plates for calibration, we are able to constrain the amount of nonstructural water in the second sanidine to less than  $0.0025 \text{ wt}\%$  (25 ppm). If structurally bound  $\text{H}_2\text{O}$  were present, the maximum concentration would be  $0.004 \text{ wt}\%$  (40 ppm).

The ammonium ion has radius and charge similar to those of K and has been observed in feldspars from pegmatites (Solomon and Rossman, 1988). There is no indication of a sharp band at  $3200 \text{ cm}^{-1}$ , which would be expected from the presence of ammonium ( $\text{NH}_4^+$ ), so we deduce that its concentration in this sample can be no more than 10 ppm.

**Kyanite.** The kyanite is also from the Roberts Victor coesite grosspydite (SRV-1) described by Smyth and Hatton (1977). The crystal selected for study is a pale blue cleavage flake, approximately  $400 \times 300 \mu\text{m}$ , which was polished on the cleavage surfaces to a thickness of  $57 \mu\text{m}$ .

Hydroxide ions were readily seen in the IR spectrum of kyanite (Fig. 1c). The spectrum of our kyanite is comparable in all aspects (except absolute intensity) to both published (Beran and Goetzinger, 1987) and unpublished (Bell and Rossman) spectra of kyanite from eclogites in South African kimberlites (Table 2). The intensity of the bands falls in the range of intensities observed in the other samples. Using the calibration of Beran and Goetzin-

ger (1987), we estimate the concentration of  $\text{H}_2\text{O}$  in this sample to be  $0.11 \text{ wt}\%$   $\text{H}_2\text{O}$  as OH.

**Corundum.** Corundum occurs as primary grains in kyanite eclogites and grosspydites in the South African eclogite assemblage, usually as inclusions in kyanite or garnet, but rarely in grain-boundary contact with clinopyroxene. Primary corundum has not been reported to coexist with coesite. A light pink single crystal from sample JIG2108 was separated and prepared for IR examination by polishing two parallel faces approximately perpendicular to  $c$  for a sample thickness of  $320 \mu\text{m}$ . The crystal was perfectly transparent with no visible inclusions. An electron microprobe analysis is in Table 1.

The IR spectrum in the region of  $3000$  to  $3800 \text{ cm}^{-1}$  is shown in Figure 1d and gives absolutely no indication of sharp band OH or of broad band molecular  $\text{H}_2\text{O}$ . Given the thickness of the sample, the maximum possible  $\text{H}_2\text{O}$  content would be  $0.4 \text{ ppm}$ , based on typical OH absorption intensities.

**Phlogopite.** The most abundant K-bearing phase in mantle eclogites, phlogopite is common as a secondary phase in most mantle eclogites from South African diamond-bearing kimberlites but is rare or absent in coesite- or corundum-bearing samples. Phlogopite may also occur as coarse-grained (up to 2 mm) veins and single-crystal inclusions in clinopyroxene. In some samples, the inclusions appear as oriented lamellae.

A transparent phlogopite crystal occurring as a hexagonal-shaped single-crystal inclusion in green Cr-rich clinopyroxene in eclogite sample SBB-102 from the Bobbejaan Mine at Bellsbank was selected for examination. A chemical analysis is given in Table 1. The sample was in a double-polished standard petrographic thin section mounted in epoxy on glass. The thickness of the section plus epoxy was measured to be  $36 \mu\text{m}$ . From a measured IR spectrum of the epoxy of known thickness, the thickness of the epoxy under the sample crystal was estimated to be  $3 \mu\text{m}$ , giving a crystal thickness of  $33 \mu\text{m}$ . The IR spectrum of the phlogopite in Figure 1e has been corrected for the glass and epoxy.

The IR spectrum in the region  $3000$  to  $3800 \text{ cm}^{-1}$  (Fig. 1e) shows a sharp feature corresponding to the presence of the expected OH in phlogopite. The spectral features of  $\text{NH}_4^+$  in mica have been previously identified as a distinctive pattern in the region  $3300$  to  $2800 \text{ cm}^{-1}$  (Higashi, 1982). This pattern is not present in our sample. Although our detection limit is somewhat compromised by the weak, broad interference fringes running through the spectrum, using the calibration data for  $\text{NH}_4^+$  in feldspar (Solomon and Rossman, 1988), we conclude that there could be no more than  $0.004\%$   $\text{NH}_4^+$  in this mica.

**Rutile.** Also a common accessory phase in mantle eclogites, rutile occurs as transparent acicular inclusions in garnet and clinopyroxene and as brown single-crystal interstitial grains up to 1 mm in greatest dimension. The larger grains typically contain small opaque inclusions, possibly ulvospinel. The larger grains also typically contain up to  $1.0 \text{ wt}\%$  each of  $\text{Cr}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{Al}_2\text{O}_3$ , and  $\text{Nb}_2\text{O}_5$ ,

**TABLE 2.** Comparison of absorbance of 3380  $\text{cm}^{-1}$  band in kyanite SRV-1 to other mantle kyanite data

Locality sample	Thickness mm	Absorbance abs units	Absorbance abs units $\text{cm}^{-1}$		Reference
			Polarized	Unpolarized	
Roberts Victor SRV-1	0.217	0.0503	2.32	1.16	This work
Roberts Victor	0.15	0.029	n.d.	1.93	Beran and Goetzinger (1987)
Roberts Victor	0.23	0.034	n.d.	1.48	Bell and Rossman (unpub.)
Reitfontein	1.317	0.11	n.d.	0.84	Rossman and Bell (unpub.)

with some grains containing up to 9.0 wt%  $\text{Nb}_2\text{O}_5$  and up to 5 wt% of the above trivalent metal oxides. It is unclear whether the material is part of the primary assemblage or of the high-pressure secondary assemblage that includes low-Na clinopyroxene, spinel, amphibole, and phlogopite.

Three of the large rutile grains in two eclogites from Roberts Victor (samples EJB-86, and SRV-16) were examined, both in epoxy-mounted thin sections. All three produced similar IR spectra, with a double sharp feature at 3320 and 3300  $\text{cm}^{-1}$ . The features are strongly polarized, being much stronger for the  $\omega$ -ray than the  $\epsilon$ -ray. Two polarized spectra from one of the samples, corrected for glass and epoxy, are in Figure 1f. The sharp features are apparently the result of significant amounts of OH in the structure.

The significant OH content of rutile was not expected and may be of some importance. Rutile is not abundant in these samples, rarely composing more than 0.5% modally of the rock. However, stishovite has the same crystal structure and may occur in the transition zone of the mantle. Little is known about the minor-element inclusion behavior of stishovite, and if it behaves like the isostructural rutile, it is likely to be much more complex than the tetrahedrally coordinated silica phases, quartz and coesite.

**Alkremite.** A prealuminous rock composed of roughly equal proportions of garnet and spinel, alkremite has been reported from several South African kimberlites. It is quite rare, so that it composes much less than 1% of all inclusions from these pipes, and it does not appear to occur preferentially in the eclogite-rich pipes at Roberts Victor and Bellsbank. It has been suggested that alkremites may represent residua from partial melting of subducted pelitic rocks in the mantle (Mazzone and Haggerty, 1988). Sample SFS-3 from the Frank Smith mine is composed of approximately 65 modal %  $\text{Gr}_{52}\text{Pyr}_{58}\text{Alm}_{20}$  garnet and 35 modal % green pleonaste spinel. Compositions of the phases are in Table 1.

**Garnet.** The garnet from sample SFS-3 alkremite shows a broad absorbance feature at 3580 to 3640  $\text{cm}^{-1}$ , which appears quite variable in intensity and shape with sample thickness and is similar to those observed for other upper-mantle garnets (Bell and Rossman, 1988). A typical spectrum from sample SFS-3 garnet is in Figure 1g. The sample contained numerous fine fractures, which were difficult to avoid in taking spectra. It appears that because of the breadth of the absorbance peak and the presence

of the fine fractures, some of the absorbance band may be due to molecular  $\text{H}_2\text{O}$  in the fractures.

**Spinel.** The composition of the spinel of sample SFS-3 (Table 1) is similar to secondary spinel occurring in many of the biminerals eclogites. An IR spectrum in the range 3000 to 3800  $\text{cm}^{-1}$  is in Figure 1h. The broad absorbance feature in this spectrum is caused by  $\text{Fe}^{2+}$ , and there is no indication of either  $\text{H}_2\text{O}$  or OH in the sample.

The absence of OH and  $\text{H}_2\text{O}$  in this spinel is an indication that this structure is less able to accommodate  $\text{H}_2\text{O}$  or OH than the coexisting garnet. Thus, spinel is an unlikely carrier of  $\text{H}_2\text{O}$  or OH in the mantle. Although spinel is not a major phase in mantle eclogite, it is a major phase in these very rare alkremite xenoliths. The absence of hydrous components in the spinel may have implications for the transition zone of the mantle from depths of 525 to 670 km, where  $\text{Mg}_2\text{SiO}_4$  adopts the structure of spinel ( $\gamma$ - $\text{Mg}_2\text{SiO}_4$ ). These results imply that unlike  $\beta$ - $\text{Mg}_2\text{SiO}_4$ , the  $\gamma$  phase is unlikely to contain significant amounts of OH, consistent with the electrostatic site potential of the O site in  $\gamma$ - $\text{Mg}_2\text{SiO}_4$  (Smyth, 1989).

## CONCLUSIONS

Coesite appears to contain virtually no  $\text{H}_2\text{O}$  or OH. The absence of OH in coesite contrasts with the association of OH in terrestrial quartz with Al substitution.

Sanidine contains virtually no  $\text{H}_2\text{O}$ , OH, or ammonia, although one crystal was observed to contain abundant  $\text{H}_2\text{O}$ , apparently as fluid inclusions of molecular  $\text{H}_2\text{O}$ . The presence of sanidine in this rock instead of phlogopite, the more common potassic phase in eclogites, is more probably caused by high silica activity in this coesite-bearing rock than by the absence of  $\text{H}_2\text{O}$ .

Kyanite appears to contain significant OH, consistent with previous results for similar samples. Using the calibration proposed by Beran and Goetzinger (1987), this kyanite has 0.11 wt%  $\text{H}_2\text{O}$  as OH.

Corundum gives no indication of the presence of  $\text{H}_2\text{O}$  or OH.

Rutile containing significant amounts of Fe, Cr, Al, and Nb also contains significant amounts of structural OH. Stishovite has the same crystal structure, and these results may indicate that minor-element and OH uptake of natural stishovite in the mantle are likely to be considerably larger than those of coesite or quartz.

Garnet in alkremite appears to contain small amounts of molecular  $\text{H}_2\text{O}$ . Spinel coexisting with the garnet in alkremite gave no indication of  $\text{H}_2\text{O}$  or OH, indicating

that the spinel structure is not able to accommodate significant amounts of OH. This may indicate that  $\gamma$ -Mg<sub>2</sub>SiO<sub>4</sub>, unlike  $\beta$ -Mg<sub>2</sub>SiO<sub>4</sub>, may be unable to accommodate OH.

Ammonium (NH<sub>4</sub>) was below detection limits (about 10 ppm) in all samples.

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